भारतीय मानक
Indian Standard

IS 1801 : 2016
(Reaffirmed 2021)

सिट्रॅनेलॉल — विशिष्टि

(चौथा पुनरीक्षण)

Citronellol — Specification

(Fourth Revision)

ICS 71.100.60

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June 2016 **Price Group 2**

Fragrance and Flavour Ingredients Sectional Committee, PCD 18

FOREWORD

This Indian Standard (Fourth Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Fragrance and Flavour Ingredients Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was published in 1961 and first revised in 1981, incorporating an additional method of gas chromatographic analysis. Subsequently in the second revision, for determining the purity as total alcohols (that is, citronellol, nerol) GLC was made the main method. The wet method of analysis was also retained as an additional method, since it was felt that citronellol is not a single compound but a mixture of various compounds and resolution by GLC may be difficult at times. The third revision was published in 1997, where the requirements for citronellol were modified taking into account that citronellol produced in the country commercially was available from any one of the natural essential oils like Citronella oil (Java type), *Eucalyptus citriodora* and/or Lemongrass oil. In fact, citronellol produced from any of those oils was not a single compound but contained, in varying amounts, other compounds such as geraniol, citronella, dimethyl octanol, *iso*-pulegol, and their esters along with traces of nerol. Accordingly the requirements of odour, relative density, refractive index were modified. New requirement of free citronellol content by GLC method and the total ester content were included.

The Sectional Committee responsible for the formulation of this standard noted that as a practice, the fragrance and flavour ingredients are traded at International level quoting the relative density and refractive index at 20 °C rather than at 27 °C as prescribed in the Indian Standards. Therefore, the Committee felt that the standard should be revised again to incorporate values for these important characteristics at 27 °C (to cater to domestic needs) and at 20 °C (to facilitate International trade).

In this revision, the gas chromatographic method given under Annex A is for guidance has been modified to include profiles through polar and non-polar columns. It is expected that the corresponding chromatograms would facilitate better identification and purity profile. The Sectional Committee also noted that as a practice, the fragrance and flavour ingredients are traded at International level quoting the relative density and refractive index at 20 °C rather than at 27 °C as prescribed in the Indian Standards. Therefore, the Committee felt that the standard should be revised again to incorporate values for these important characteristics at 27 °C (to cater to domestic needs) and at 20 °C (to facilitate International trade).

Citronellol ($C_{10}H_{20}O$) is one of the most important perfumery raw materials, widely used in various fragrances and is also used as an effective sweetener in floral and fancy formulations.

Citronellol is found in a wide variety of essential oils, mainly, Citronella oil (Java type) and geranium. Citronellol ex-geranium oil is laevorotatory and is called ‘Rhodinol’ in trade. However, citronellol produced in India and commercially available, is optically dextrorotatory and is produced by the catalytic reduction (hydrogenation) of citronellal present in the Citronella oil (Java type) or *Eucalyptus citriodora* or the Citral present in the Lemongrass oil.

Citronellol is also synthesized from alpha-pinene and/or beta-pinene but this is not commercially produced in the country. It should be borne in mind that today the over whelming bulk of citronellol produced in the world is from alpha and/or beta-pinene. In India, even though no production takes place through this route, the major quantity of citronellol consumed is that which is produced ex-alpha and/or beta-pinene, the requirements being met through imports.

(*Continued to Third Cover*)

Indian Standard
CITRONELLOL — SPECIFICATION
(Fourth Revision)

1 SCOPE

This standard covers the requirements and methods of sampling and test for citronellol commercially produced from Citronella oil (Java type) and *Eucalyptus citriodora* oil.

2 REFERENCES

The standards listed below contain provisions which, through reference in text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All the standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of these standards.

IS No.	*Title*
326	Methods of sampling and test for natural and synthetic perfumery materials:
(Part 1) : 1984	Sampling (*second revision*)
(Part 2) : 1980	Preliminary examination of perfumery materials and samples (*second revision*)
(Part 3) : 2006/ ISO 279: 1998	Determination of relative density (*third revision*)
(Part 4) : 2005/ ISO 592 : 1998	Determination of optical rotation (*third revision*)
(Part 5) : 2006/ ISO 280 : 1998	Determination of refractive index (*third revision*)
(Part 6) : 2005/ ISO 875 : 1999	Evaluation of miscibility in ethanol (*third revision*)
(Part 8) : 2005/ ISO 709 : 2001	Determination of ester value (*third revision*)
(Part 9) : 1980	Determination of ester value after acetylation and free alcohols (*second revision*)
(Part 11) : 1986	Determination of carbonyl value and content of carbonyl compounds (*second revision*)
(Part 19) : 1998	Gas chromatographic analysis of perfumery materials
1070 : 1992	Reagent grade water — Specification (*third revision*)
2284 : 1988	Methods for olfactory assessment of natural and synthetic perfumery materials (*first revision*)
6597 : 2001	Glossary of terms relating to fragrance and flavour industry (*second revision*)

3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 6597 shall apply.

4 REQUIREMENTS

4.1 The material shall be a clear liquid free from sediment, suspended impurities, separated water and adulterants and shall be examined by the method prescribed in IS 326 (Part 2).

4.2 Solubility

The material shall be soluble in two volumes of ethanol (70 percent by volume) when tested as prescribed in IS 326 (Part 6).

4.3 The material shall also comply with the requirements given in Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be supplied in air-tight amber coloured bottles or opaque containers such as aluminium containers or steel drums which are expoxy lined, permitting a minimum of air space as agreed to between the purchaser and the supplier.

5.1.1 The material shall be protected from light and stored in a cool and dry place.

5.2 Marking

The material shall be marked with the following information:

a) Name of material;
b) Name of the manufacturer and his recognized trade-mark, if any;
c) Net mass of the material;
d) Batch number; and
e) Year of manufacture.

5.2.1 The containers may also be marked with the Standard Mark.

Table 1 Requirements of Citronellol

(*Clauses* 4.3 *and* 7.1)

Sl No. (1)	Characteristic (2)	Requirement (3)	Method of Test, Ref to IS No. / Annex (4)
i)	Colour and appearance	Colourless clear liquid	326 (Part 2)
ii)	Odour	Fresh floral rose note and free from grassy and/or minty by-odours	2284
iii)	[1]Relative density at 27/27°C	0.848 to 0.859	326 (Part 3)
iv)	[1]Refractive index at 27°C	1.451 to 1.460	326 (Part 5)
v)	Optical rotation	–1° to + 5°	326 (Part 4)
vi)	Total alcohols, content calculated as citronellol, percent by mass, *Min*	96	326 (Part 9)
vii)	Free citronellol content, percent by mass, *Min*	88	Annex A
viii)	Total carbonyl content (inclusive of citronellal/citral), percent by mass, *Max*	0.5	-do-
ix)	Total esters content[2], percent by mass, calculated as citronellyl acetate[2] (molecular weight 198.31), *Max*	1.0	326 (Part 8)

[1] The correction factor for relative density and refractive index for each degree Celsius change in temperature is 0.000 64 and 0.000 38 respectively.

[2] The refluxing time is 1 h [*see* **9.3.1** IS 326 (Part 8)].

5.2.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. Details of conditions under which a licence for the use of the Standard Mark may be obtained from the Bureau of Indian Standards.

6 SAMPLING AND CRITERIA FOR CONFORMITY

6.1 Representative samples of the material shall be drawn as prescribed in IS 326 (Part 1).

6.2 All the characteristics given in this standard shall be tested on composite sample. The lot shall be declared as conforming to the requirements of this standard if all the results on the composite sample meet the corresponding requirements.

7 TESTS

7.1 Tests shall be carried out as prescribed under **4.1, 4.2** and the appropriate references specified in col 4 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (vii) *and* (viii)]

ANALYSIS OF CITRONELLOL

A-0 GENERAL

For determining total alcohols, free citronellol and total carbonyl content, GLC method has been prescribed as the main method. In specific cases where difficulty is experienced in resolving mixture of components by GLC, citronellol content and total carbonyl compounds may be determined as specified in IS 326 (Part 9) and IS 326 (Part 11) respectively.

NOTE — For determination of carbonyl compound, 5 g of sample may be taken and preceded by cold hydroxylamine method. For ester determinations, 5 g of the sample may be taken for the test.

A-1 GAS CHROMATOGRAPHIC ANALYSIS FOR CITRONELLOL

A-1.1 The chromatographic conditions given here are for information and guidance only.

A-1.2 Outline of the Method

A sample of the material is dissolved in a suitable solvent such as, cyclohexane or diethyl ether or acetone and is injected into the gas chromatograph, from where it is carried by the carrier gas from one end of the column to the other. During its movement, the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column.

A-2 PROCEDURE

A-2.1 The analysis shall be done as per IS 326 (Part 19). The typical chromatogram for citronellol in capillary column with the following chromatographic conditions is shown in Fig. 1 and Fig. 2.

A-2.1.1 *Gas Chromatographic Conditions on Polar Column*

Column and phase	Fused silica polar column[1]
Film thickness	0.25 μm
Column dimensions	30 m × 0.25 mm ID
Sample size	0.5 μl split 1:200
Carrier and flow	30 ml Helium per min, velocity 22.9 cm/s, flow control mode
Injector temperature	240°C
Detector type	Flame ionization detector
Detector temperature	260°C
Oven temperature	160°C
GC temperature program	4°C per min upto 210°C

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[1] SUPELCOWAX 10

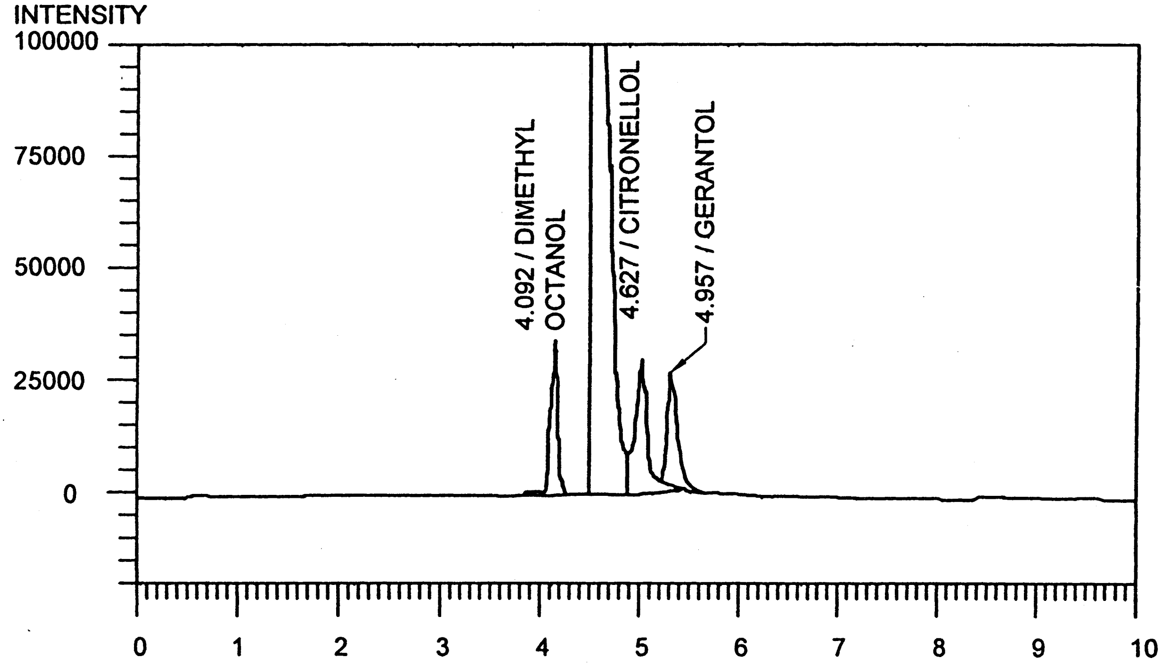

FIG. 1 CITRONELLOL — GC PROFILE ON POLAR COLUMN

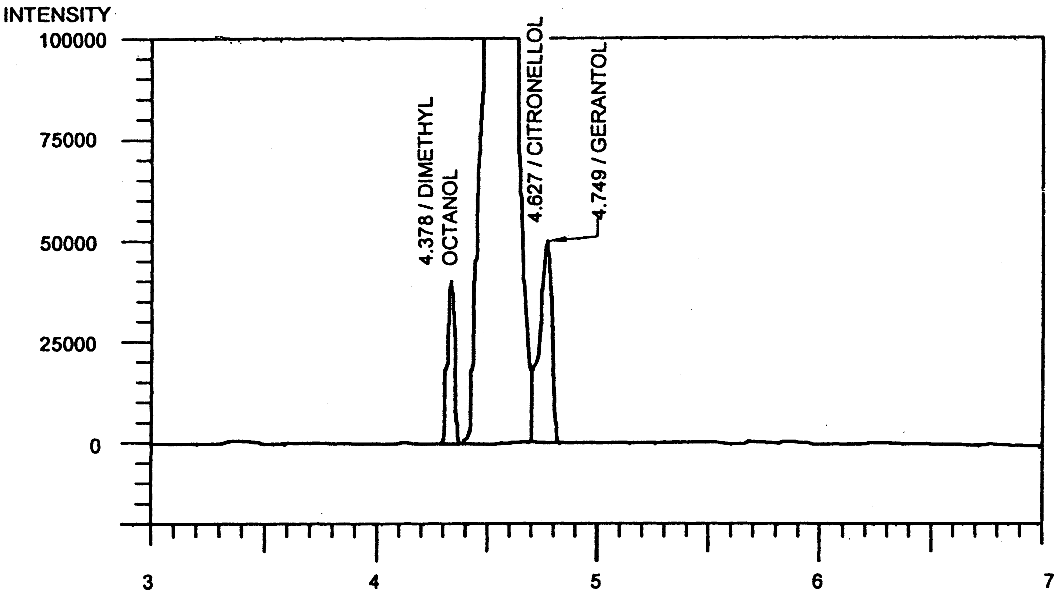

FIG. 2 CITRONELLOL — GC PROFILE ON NON-POLAR COLUMN

A-2.1.2 *Gas Chromatographic Conditions on Non-Polar Column*

Column and phase	Fused silica coated with non-polar phase[1]
Film thickness	0.25 µm
Column dimensions	30 m x 0.25 mm ID
Sample size	0.5 µl split 1:200
Carrier and flow	30 ml Helium per min, velocity 22.9 cm/s, flow control mode
Injector temperature	240°C
Detector type	Flame Ionization Detector
Detector temperature	260°C
Oven temperature	160°C
GC temperature program	4°C per min upto 210°C

A-3 CALCULATION

A-3.1 Area Measurement (*see* Note 1)

Since normal peaks approximate a triangle, the area is measured by multiplying the peak height with the width of half-height. The normal peak base is not taken since large deviations may be observed due to tailing or adsorption. This technique is rapid, simple and fairly accurate when peaks are symmetrical and of reasonable width.

A-3.2 Area Normalization (*see* Note 2)

By normalizing, it is meant, calculating the percentage composition by measuring the area of each and dividing the individual areas by total area, for example:

$$\text{Percentage of } a = \frac{\text{Area of } a}{\text{Total Area}}$$

NOTES

1 Other methods of area measurements, namely, triangulation, disc integrator and electronic digital integrator, if fixed with GLC machine, would be of great advantage.

2 Internal standardization may be used if pure appropriate internal standard is available. This method is relative or indirect calibration.

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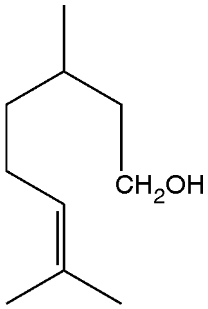
[1] Methyl silicon EQUITY-1.

(*Continued from Second Cover*)

It is represented by the following structural formula:



3, 7-DIMETHYL -6-OCTENE-1-OL
(MOLECULAR MASS 156.27)

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Bureau of Indian Standards

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This Indian Standard has been developed from Doc No.: PCD 18 (2356).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi